Abstract

The design of electrode, device architecture, and functional properties of the material play an important role in fabrication of energy devices and sensors. The literature is bound with various examples of nanomaterials including metal oxides and spinels as electrocatalysts in energy-efficient systems and sensing applications. The conventional methods for synthesizing these nanomaterials are well established; however, they often fail to address the stability, durability, reproducibility and scalability issues that are stringent requirements for practical application. In this prospect, the large-scale fabrication of electrocatalytic materials with controllable and flexible preparation remains a challenge since various processes are multistep, instrumentation-dependent and often result in loss of activity during synthesis. For commercial applications, it is interesting to synthesize solution-processable inks that can be coated on the choice of conductive support system for the fabrication of active electrodes. In this thesis work, Ni and Co-based alkanethiolate complexes with lamellar structure are used as functional inks that could be directly coated on the desired substrates, and the processing conditions are rigorously optimized for application as electrocatalyst in oxygen evolution reaction (OER), development of glucose sensors, biomolecular sensors, chemical sensors and as a catalyst in the synthesis of graphite for the fabrication of supercapacitors.

A brief outline of the thesis work is as follows:

The **first chapter** provides a general introduction and literature survey to the overall work carried out in the thesis, while the **second chapter** provides a detailed account of experimental techniques used for the synthesis, characterization, and data analysis.

In the **third chapter**, Ni alkanethiolate complexes with different alkyl chain lengths are electrooxidized for the fabrication of electrodes for glucose sensing. The Ni butanethiolate (Ni-BT) with the shortest chain length resulted in more sharp redox peaks after surface electrooxidation. It is further coated on transparent and conducting Au mesh electrodes and used as a test platform for enzymeless glucose sensing with a high sensitivity of 675.97 $\mu A.mM^{-1}cm^{-2}$ and detection limit of 2.2 μM along with excellent selectivity and reproducibility.

In **chapter four**, Co Hexadecanethiolate (Co-HDT) and Ni-BT are coated on carbon cloth substrates by layer-by-layer (LbL) dip-coating method and thermolysed to form Co_3O_4 and NiO nanostructures as electrocatalyst for the OER process. The importance of LbL deposition of Co-HDT and Ni-BT ink was studied by comparing the dip-coating of the highest concentration and multiple LbL dip-coating in dilute solutions. The LbL dip-coated electrodes possessed remarkable stability over 24 hours with an overpotential of 300 and 340 mV at 10 mA/cm^2 and Tafel slope of 77.06 and 65.5 mV/dec, respectively.

In **chapter five**, the Co-HDT and Ni-BT were chosen as inks to form mixed lamellar bilayer assemblies at an optimized ratio of 4:1 respectively, that give rise to interesting bimetallic Ni-Co spinel nanoplates by solventless decomposition method. Electrocatalyst formed by this method exhibited excellent electrocatalytic activity towards oxygen evolution reaction (OER) and is studied by XPS measurements. The influence of magnetic field on OER performance of superparamagnetic nanoplates of NCO is further explored in this study.

In **chapter six**, $NiCo_2O_4$ nanoplates are explored for simultaneous detection of various biomolecules such as dopamine and uric acid with high sensitivity in a wide working range. The dopamine and uric acid exhibited a LOD in nM range which lies in the desired ranges of detection.

In **chapter seven**, the Ni-BT ink is used as a catalyst in ultralow quantities for conversion of waste polystyrene to graphitic nanocarbon under an optimized temperature (800 °C) in the presence of 5% hydrogen in nitrogen for application in EDLCs. The graphitic carbon electrode exhibited perfect rectangular CV characteristics, symmetric triangular charge-discharge curves (C_{sp}~158 F/g at 1 A/g), excellent cyclic stability, and high capacitance retention of ~ 90% even after 10,000 cycles.

The **eighth chapter** presents the conclusion and some thoughts of the future work.